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INVESTIGATION OF THE PROPERTIES OF METALS AND ALLOYS AT HIGH TEMPERATURES IN VACUO

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[Figures referred to are appended.]

Microscopic analysis of metals and alloys on heating is of great scientific and practical significance since it permits establishment of the relation between temperature and changes in structure and physicochemical state of the systems under investigation. Especially, it is essential for numerous heat-resistant alloys which have lately received quite extensive industrial application.

First works in the field of developing methods for investigating the structure of metals and alloys at high temperature were conducted by A. A. Baykov in 1909 [1]. For studying the polymorphous transformations of iron, Baykov used the etching of the polished surface of specimens heated to a prescribed temperature. Heating was conducted in a hydrogen atmosphere to prevent oxidation of the specimen surface by oxygen in the air. However, the appearance of specimens was modified under the action of hydrogen on the polished surface: they acquired a silvery coloration distinct from the original natural color.

Experiments for heating specimens in other gas media gave no satisfactory results. It was established that new methods must be found for studying the structure of metals in a heated state. Baykov decided to try heating and etching of specimens in vacuo. First experiments, conducted on an installation designed by N. T. Gudtsov [2], gave valuable results: microscopic examination of specimens revealed the basic structures of polymorphic modifications in iron. In addition, it was possible to fix structural modifications, which take place at moments of transformations in iron and in tool carbon steel of eutectoid and hypereutectoid composition in the range from 600 to 1,100°C. These results were obtained for the first time in the science of metals.

In 1915, N. Chizhevskiy and N. Shul'gin [3], attempted to reveal the structure of alloys by etching specimens with chlorine after heating in vacuo.

A series of investigations was conducted in 1937 by L. I. Shushpanov [4] in the Leningrad Industrial Institute. He studied the behavior of the polished surface of specimens of seven various steels under heating in vacuo without etching and established that a single heating in vacuo above the temperature of phase transformations is sufficient for stabilization of polyhedral structure.

The work by K. L. Malyshev and I. B. Trubin [5] gives a description of experiments for revealing the boundaries of grains in various types of steel by heating the microscopic specimens in vacuo. The intensive evaporation from the grain boundaries, which occurs on the surface of specimens of carbon steel at temperatures above 1,000° in a vacuum, permits clear revelation of the sizes of austenitic grains. An increase of temperature is required for revealing the structure of alloy steel. For example, satisfactory results for chromium-nickel machine steel were obtained only at 1,050°.

Work on the study of the properties of metals and alloys at high temperatures has been conducted under the supervision of Academician N. T. Gudtsov in the Institute of Metallurgy imeni A. A. Baykov of the Academy of Sciences USSR for several years. This paper gives a description of equipment developed by M. G. Loginskiy and N. A. Bogdanov and also presents examples of investigations conducted by the authors.

Vacuum Installation for Experimental Works

1. Method of Heating the Specimens

The method of heating the specimens in vacuo is one of the essential factors which have an effect on the structural form of the device for revealing the structure of metals and alloys at high temperature. Three heating methods were considered: in a quartz tubular furnace, by induction, and the contact method of heating a specimen by means of passing an alternating current of industrial frequency. The contact electric heating has many advantages in comparison with other methods; for example, it makes possible regulation of the rate of temperature increase over a wide range: from a small fraction of a degree per minute to tens of thousands of degrees per minute. In addition, heating of samples by electric current permits obtaining any temperature up to and including melting.

The electric heating of specimens up to 25 sq mm in cross-sectional area was realized by a single-phase 6-kw welding transformer with primary voltage of 220 v and secondary voltage up to 10 v. The current passing through a specimen could be regulated over a range from 100 to 3,000 amp with the aid of a 12-step autotransformer. A 4-kw autotransformer with a variable transformation ratio was used for smooth change of current.

The system described made it possible to heat the specimens at any prescribed rate and also permitted holding them at a definite temperature during experiments.

2. Method of Measuring Temperature on the Surface of a Specimen

The most precise control of temperature on the surface of the specimen under investigation is essential. Several methods for measuring temperature in vacuo were tried. Visual control of temperature with optical pyrometers and ardometers was excluded since evaporation from the surface of steel specimens begins at temperatures above 1,000° and condensation of metal vapors on a sight glass considerably alters the readings of pyrometers.

Temperature control was realized with the aid of thermocouples welded to separate parts of a specimen. At temperatures up to 800°C chromel-alumel thermocouples were used and platinum versus platinum-rhodium thermocouples were employed in the investigation of specimens heated to higher temperatures.

The thermocouples were welded to specimens by the method of resistance spot welding with a current supplied from the same transformer which produced the current for heating. The device developed by the authors provided for connection of as many as five thermocouples welded to various zones of a sample. This secured determination of the temperature gradient along a specimen. Numerous measurements demonstrated that the welding of thermocouples to a specimen secures an inertialess control of temperature on the investigated surface.

3. Vacuum System of the Experimental Installation

Figure 1 presents a schematic diagram of the vacuum system of the experimental installation for investigating the properties of metals and alloys at high temperatures. The numeral 1 denotes a bell made of molybdenum glass. The inside diameter is 250 mm and the height is 320 mm. This bell serves to hold the specimen with thermocouples and the devices for studying the intensity of evaporation and for hardness measurement in vacuo. The edge of the bell is thoroughly ground to a steel plate 25 mm thick. The edge of the bell and corresponding surface portion of the plate (2) have to be coated with vacuum grease -- a solution of natural rubber in viscous vaseline.

Two pipes of 22-mm diameter are connected to the steel plate. A vacuum cock (3) permits connection of the space under the bell with an evacuating system. A mercury U-shaped vacuum gauge (4) serves for observation of the evacuation rate and approximate determination of the degree of vacuum under the bell. A dual system of evacuation with diffusion and rotary pumps is employed for obtaining a high vacuum to 10^{-6} mm Hg. A glass trap (5) protects the system against penetration of oil vapors from a metal diffusion steam-oil pump (6). The productive capacity of the pump is about 15 liters/sec. A forevacuum tank 7 of 120 mm diameter and 400 mm length is installed between the pump (6) and cock (8). The cock (8) permits disconnecting the diffusion pump from the zone of heating and etching of specimens. This disconnection is quite necessary since, otherwise, the vaseline oil in the diffusion pump may be oxidized and contaminated by penetration of various chemically active substances used for etching the specimens. A glass cock (9) is designated for establishing atmospheric pressure in the system. A trap (10) serves to prevent oil penetration into the system from the rotary pump (11) in case it stops when the cock (9) is closed, as may happen during an emergency switching off of supply voltage and the stopping of the electric motor (12) which rotates the pump. The pump (11) has a capacity about 55 liters/min and provides for evacuation to 10^{-3} mm Hg.

Two vacuum gauges are used for measuring the residual pressure: a thermoelectric junction instrument and an ionization type vacuum gauge. The thermoelectric junction vacuum gauge utilizes a special tube (13) of type L T-2. This tube has a thin platinum wire to which the thermojunction of an iron-constantan thermocouple is welded. Direct or alternating current of 125-135 ma is conducted through the platinum wire. The performance of the vacuum gauge is based on variations in the resistance of the platinum wire depending on pressure. The temperature of the wire rises with a decrease of residual pressure. The value of thermoelectric current in the L T-2 tube permits evaluation of the degree of vacuum in the system in the range from 0.1 to 10^{-4} mm Hg. A cock (14) makes possible disconnection of a IM-2-type tube (15), which is included in an ionization vacuum gauge of the VI-1 type. The latter permits measurement of residual pressures from 10^{-3} - 10^{-7} mm Hg.

The chemically active medium required for etching heated specimens is introduced into a space under the bell with the aid of the following device. Vacuum cocks (16) and (17) are installed on the outgoing pipes of a measuring vessel (18), approximate volume of which is 50 cu cm. The pipe portion (19) is filled with calcium chloride which prevents contamination of oil in the pump (11). The pressure of an etching medium is measured by a MacLeod-type mercury vacuum gauge (21) connected to the system by way of a ground joint (20).

A special "sluice" device, consisting of two stopcocks (22) and (23), permits introduction into the system of definite amounts of gases, such as hydrogen chloride, hydrogen fluoride, nitrogen oxides and others, from a tank (24). The portion of the pipe between the cocks (22) and (23) is filled with gas when the cock (22) is closed and (23) is open. After closing the cock (23) and opening (22) the amount of gas contained in the pipe between (22) and (23) is introduced into the system. The pressure in the system is measured with the vacuum gauge (21) and may be decreased by evacuation through the cock (17) or increased by introduction of another batch of gas.

Constructional arrangement of separate elements of the vacuum system is shown in Figure 2. Parts enumerations 1 to 24 are the same as in Figure 2. Number (25) denotes the hollow water-cooled electrodes insulated from the plate (2). Six terminals (26) are used for connecting the thermocouples welded to the specimen. The temperature is measured by readings from galvanometer (27), equipped with a change-over switch for connection with separate thermocouples. An ionization vacuum gauge (28) of VI-1 type is located next to the galvanometer. Over-all dimensions of the assembly are 725 x 1,030 mm and 1,455 mm high.

The installation described is designated for heating specimens to high temperatures, including melting, and permits study of the structure of metals and alloys either by the method of etching the heated polished surface with various aggressive gas media or by observing the phenomenon of metal evaporation from the separate zones of the polished surface.

4. Shape of the Specimens for Studying the Structure on Heating in Vacuo

Specimens of various shapes were made in the process of conducting the experiments for revealing the structure of metals and alloys at high temperature.

The most expedient shapes for specimens are presented in Figure 3. The wedge-shaped specimen is shown in Figure 3, a. An advantageous feature of these specimens is the possibility of obtaining any temperature gradient along the length of a specimen. The amount of heat liberated in separate zones of a sample is proportional to the square of current and to the resistance of a given part of a specimen. In the case of varied cross section of a specimen, the resistance is a value inversely proportional to the cross-sectional area. Maximum temperature will be obtained during the flow of current in parts with a minumum cross section. Application of such specimens considerably accelerates study of the structure, since a single specimen makes it possible to obtain a great variety of temperatures.

The distance between centers of the current supply electrodes is 62 mm. Therefore, holes 9 mm in diameter must be drilled in the specimens with a 62-mm distance center to center. A maximum cross section of 25 sq mm was selected with the aid of the 6-kva transformer. This area allows the zone under study to be heated to the steel's melting point.

Figure 3, b shows another shape of specimen made of a strip 2 mm thick and up to 15 mm wide. Such a shape considerably facilitates polishing of the projecting zone.

Flat specimens as shown in Figure 3, c were also used. Only one narrow side of these specimens was polished. Simultaneous polishing of several specimens is thus possible by holding all specimens in a single clamp.

To eliminate warping of specimens, as a result of their elongation on heating, a special system of fastening was employed. A specimen is attached to one electrode only with a screw on the electrode's upper end. The screw on the top of the other electrode attaches to a flexible current-conducting strip made of copper foil. Free ends of the specimen and copper strip are connected to each other with a third screw. The glass bell has a comparatively large surface of about 3,000 sq cm, which makes possible the prolonged holding of specimens in a heated state, since the specific thermal load on the glass is insignificant.

Study of the Structure of Alloys on Heating in Vacuo

Investigations showed that exposure of the microstructure on the polished surface of specimens heated in vacuo may be realized by various methods. On heating the steel specimens to 700°, an aggressive medium, reacting differently with separate structural components of steel, must be introduced into the vacuum. Hydrogen chloride, vapors of nitric and hydrochloric acids, nitrogen oxides, and air were used in this capacity.

The microstructure of specimens, heated to temperatures in the range from 700 to 900°, may be revealed either under action of an aggressive gaseous medium or simply by heating in vacuo.

The heating temperature, corresponding to the appearance of grain boundaries on the polished surfaces, will vary depending on the composition of the steel. This temperature is of the order of 800-850° for plain carbon steel. In the case of alloy steel, the temperature for the appearance of grain boundaries is 900° or higher. This phenomenon may be explained as follows.

At temperatures up to 800-850° the appearance of grain boundaries in carbon steel has no relation to evaporation of the metal itself and only depends on the evaporation of various impurities: oxides, nonmetallic inclusions, and gases, occurring along the grain boundaries and having a greater vapor tension than that of the metal. High-alloy steel has less impurities located on the grain boundaries and, therefore, the noticeable appearance of grain boundaries takes place only at temperatures which are close to the evaporation temperature of the metal itself.

The microstructure of any steel may be clearly revealed by heating polished specimens in vacuo to temperatures above 1,000°. Heating to temperatures in the vicinity of melting is of particular interest. A study of the microstructures, obtained in the case of specimens heated to the liquid-solid transition state, may permit establishment of new regularities in the process of the formation of the liquid phase and in the kinetics of primary crystallization.

1. Etching in Vacuo for Revealing the Microstructure of a Specimen in a Heated State

The experiments for revealing the microstructure of various steels were initiated by introducing certain amounts of air under the bell of the experimental installation. It was found that the modification in pressure inside of the bell from 10^{-5} mm Hg to several millimeters by admitting the air permitted a clear outlining of grain boundaries.

Separate structural components react differently with the oxygen of air; for example, perlite in the period just before transformation near point Ac_1 (moments of the greatest chemical activity) is oxidized more intensively, creating sharp visibility of the microstructure of steel. A dense layer of oxide covers the surface of specimens with an increase in the amount of air admitted.

Introduction of various gas media into the etching zone of the apparatus makes it possible to study the corrosion resistance and kinetics of the formation process of oxide films.

Application of pure chlorine and hydrogen chloride for etching the microspecimens did not give satisfactory and dependable results. A microscopic image of the etched zone was not sufficiently clear and changed in the course of time. The greatest effect was attained by etching with nitrogen oxides. The mixture of nitrogen oxides, admitted into the zone between stopcocks 22 and 23 (Figure 1) has to be released into the vacuum system by opening cock 22 at the moment when a specimen is ready for etching. Exposure of the structure with nitrogen oxides gave quite satisfactory results.

2. Utilization of the Surface Evaporation of Metals for Revealing the Structure on Heating to High Temperatures

On studying the structure of metals and alloys in a heated state at atmospheric pressure, the phenomenon of surface evaporation of metals usually is not taken into consideration. The intensity of evaporation increases sharply on heating in vacuo.

The phenomenon of evaporation of metals from the surface of microspecimens may be utilized in metallography as a new method of investigation. It is quite evident that the stronger the bond among metal atoms in the crystal lattice, the more difficult it is for atoms to overcome the forces which keep atoms together. Separation of atoms requires an increase of vacuum or temperature. Predetermination of the relation between the evaporation intensity and heat-resistant properties is difficult for the time being, but it may be assumed that a certain relation exists.

Investigators have noted that the structure of alloys appears sufficiently clear in a vacuum on heating to a high temperature, above Ac_3. This was indicated, for example, in the work by L. I. Shushpanov [4]. Various authors offer different theories which relate this phenomenon to evolution of gases from the surface of metals, anisotropy of metals, or to a tendency of the crystals to assume a spherical shape under heating to a high temperature, etc.

In the authors' opinion, structure is revealed on heating a specimen in vacuo only because of the evaporation of metals and impurities. This was corroborated by a series of special experiments. The phenomenon of evaporation can by no means be related to the critical points of steel, e.g., Ac_3. The YalT steel of austenitic type has no transformations on heating; however, it begins to evaporate intensively after reaching a definite temperature. Three parameters are the fundamental factors here: the vacuum degree, the heating temperature, and the vapor tension of the metal or impurity. In addition, it is known that the same metals have sharply varied values for evaporation rates and vapor tension depending on various experimental methods and also on the influence of impurities and contamination of the surface of the evaporating metal. Invisible films of oxides may sharply decrease the rate of evaporation from open metal surfaces. At the same time, the impurities included in the evaporating metal may contribute to evaporation [6]. For example, pure iron, according to some data from technical literature, begins to evaporate noticeably at 1,000°, although the evaporation rate of iron at this temperature is very low.

Measurement of evaporation rates was conducted by Langmuir, who also expressed it by an equation: $m = P\sqrt{\frac{M}{2\pi RT}}$

where m is the evaporation rate in g/sec, P is pressure in dynes per sq cm, M the atomic weight of the metal, R is the gas constant $8315 \cdot 10^7$ erg/g · atom, T is absolute temperature.

The experiments conducted by the authors demonstrated the possibility of revealing the grain boundaries and structure within a grain of steel by heating microspecimens in vacuo to temperatures not lower than 900-1,000°.

An increase in heating temperature causes more intensive evaporation from the surface of a specimen, as a result of which the boundaries of austenitic grains and the structure within grains became more distinct.

A device designed by the authors permits study of the deformation processes in alloys on heating. For this purpose, the specimens under investigation must be subjected to tension or torsion. Additional investigations have yet to be conducted on the use of the technique of vacuum heating for studying the strength properties of metals and alloys.

Application of contact electric heating permits raising the temperature of specimens to any value, including the melting temperature. This facilitates investigations which are very essential for studying the complex processes occurring in metals during their transition from the solid state to liquid and also during their crystallization.

Thus, the microstructure of the specimen, made of stainless chrome-nickel steel and heated in vacuo to a temperature near melting, revealed a beginning of the formation of the liquid phase in certain portions of a single grain. It may be assumed that the phenomenon of selective melting has a direct relation to the chemical heterogeneity within austenitic grains. The microstructure of a specimen brought to melting in vacuo shows dendrites formed during crystallization of a molten zone.

Device for Determining the Intensity of Evaporation on Heating Metals in Vacuo

A condensation method was used by the authors for studying the evaporation intensity of steel specimens. The layout of the device is given in Figure 4. A steel plate (1) is attached rigidly to one electrode. The arm (2) may be shifted along a slot in its left end and fixed in any position with a stop screw (3). The movable arm (4) may swing on the pin (5). A steel end plate (6) is attached to the arm (4) with the aid of a screw (7) so that the space between the plate and glass bell (8) will be approximately 1 mm. A permanent magnet or electromagnet is used for turning the arm (4) in the vacuum. The magnet acts on the plate (6) through the glass wall (8) which is not an obstacle to magnetic force lines. The arm (4) swings on its pin with motion of the magnet along the wall of the glass bell.

The condenser (11) is placed under a square cover (10) attached to the plate (1). Plates 20 x 20 mm made of glass 1.5 mm thick and mica 0.3 mm thick were used for the condenser. Figure 4 shows the position of the device when the protruded portion of the arm (4) covers the 10 x 10-mm-square opening in the plate (1). The device has to be installed in the bell in such a manner that the opening in the plate (1) is located exactly over the heated zone of the specimen. The specimens illustrated in Figure 3b were used in experiments for determining the evaporation intensity. The distance between the condenser and specimen amounted to 8-10 mm. On turning the arm (4), the condenser portion over the specimen becomes uncovered and vapors of metal begin to condense on the surface of that portion. Figure 4 shows the position of separate elements of the device described during condensation of vapors on the condenser (11). Pressing of the cover (10) to the plate (1) is possible by using screws (12) and (13).

The experiments conducted for investigat'ng the evaporation intensity of certain alloys demonstrated that heat-resistant steels evaporate less intensively than the structural steels heated to the same temperature. The metal deposited on the condenser has a crystal structure the character of which may be determined by X-raying.

It should be noted that further investigations in the study of the evaporation intensity on heating, evidently, will help to establish the relation between evaporation and the heat resistance of an alloy.

Hardness Testing of Specimens Heated in Vacuo

For hardness tests on heating to 900° in vacuo, the authors designed the device shown in Figure 5. The numerals (1) and (2) denote the current supplying electrodes; (3) is a specimen attached to the electrode (1) and the flexible damper (4) is made of copper foil. A fastener (5) is installed on the electrode (1). The cylindrical rod (6) may be fixed in required position by the fastener (5). The head (7) is connected with a bracket (8) which rotates around a vertical axis and may be kept in fixed position with a stop screw (9).

Determination of hardness is realized by measuring the indentation obtained after impressing the indenter (10) which has a tip made of pobedite with an included angle of 136° between opposite faces. Invar, the alloy with a low coefficient of thermal expansion, may be suggested as the best material for the body of the indenter. A steel ball (11) is pressed into the top of indenter. The lever (12), turning on a horizontal axis in the bracket (8), presses against the ball of the indenter. For increasing pressure on the indenter, the lever (12) may be loaded with the weights which have a stop screw for fixing them at a definite position on the lever. The quartz rest (13), 10 mm in diameter, prevents bending of the specimen under pressure from the indenter. The rest is set in a steel bar (14), which has its threaded lower portion screwed into the metal base (15). A lock nut (16) fixes the rest (13) in the required position. A fastener (17) on the electrode (2) supports a vertical rod (18) with a plate (19) attached to its top end. A dial indicator (21) is connected to the plate (19) with the aid of an insulating bushing (20) made of hard rubber.

The entire device described is enclosed in the glass bell (22) ground to the steel base plate (23). Evacuation of the space under the bell is done through the outlet (24) connected with the vacuum system.

The heating temperature is measured with a thermocouple welded to the edge of the specimen. The thermocouple is not shown in Figure 5.

The dial indicator may be used for determination of creep: the speed of penetration of the indenter into a specimen at a given temperature may characterize the heat-resistant properties of the alloy under study. Elaborate investigations have yet to be conducted in this field.

A hardness test with the aid of the device described above is similar to a Vickers hardness test. The pyramid indenter is forced into the specimen heated to a prescribed temperature. The hardness number H is the load P (kg) divided by the impression area F:

$$H = \frac{P}{F} = \frac{P}{\frac{b^2}{2\cos 22^\circ}} = \frac{P \cdot 1.854}{b^2} \text{ kg/sq mm}$$

where b is the diagonal of the square impression in mm, measured through a microscope fitted with an ocular micrometer.

Hardness testing results for three types of steel are presented in the following table:

Type of Steel	Test Temp (°C)	Load (kg)	Diagonal of Impression (mm)
Valve austenitic steel	700	0.8	0.11
	800	0.8	0.17
	900	0.8	0.23
Stainless Chrome-nickel steel	700	0.8	0.19
	800	0.8	0.23
	900	0.8	0.28
Carbon eutectoid steel	700	0.8	0.38
	800	0.8	0.5
	900	0.8	0.55

A graph illustrating the trend of changes in hardness of the same steels on heating in vacuo to 900° is presented in Figure 6. This graph permits assumption of the existence of a temperature coefficient of hardness specific to various types of steel.

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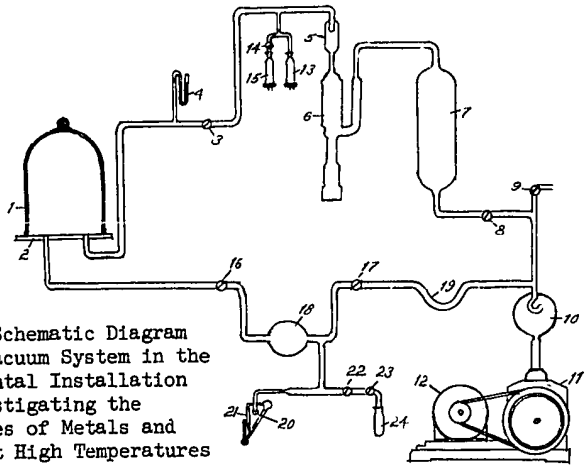

Figure 1. Schematic Diagram of the Vacuum System in the Experimental Installation for Investigating the Properties of Metals and Alloys at High Temperatures

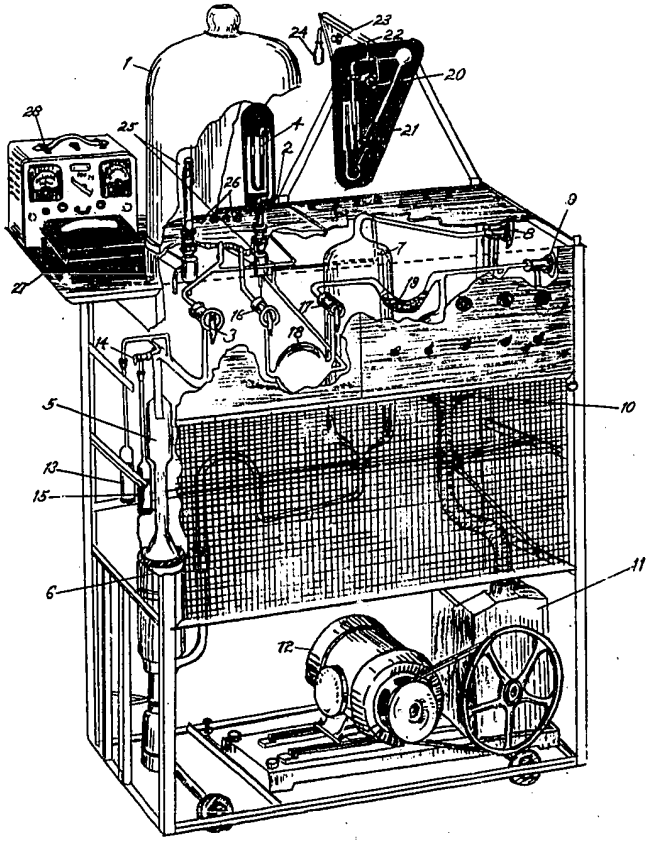

Figure 2. Constructional Arrangement of Separate Elements of the Experimental Installation for Investigating the Structure of Metals in Vacuo

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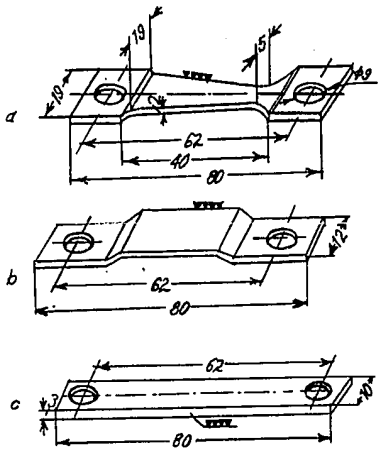

Figure 3. Shapes of Microspecimens Used for Studying the Structure of Metals and Alloys on Heating in Vacuo

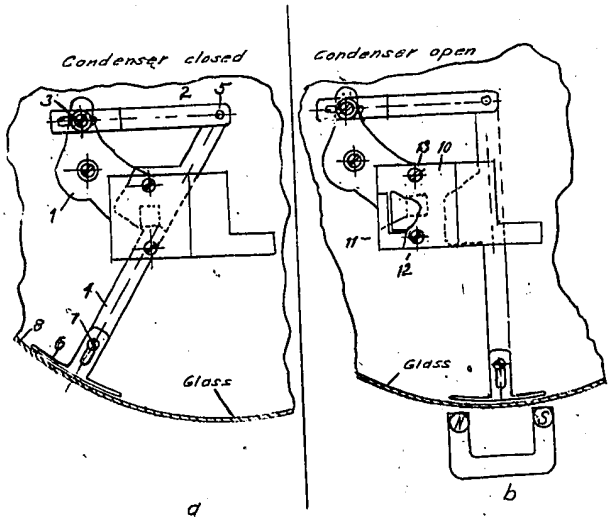

Figure 4. Layout of the Device for Studying the Evaporation Intensity of Metals in Vacuo by the Condensation Method /Numeral 9, Figure b, missing from original drawing./

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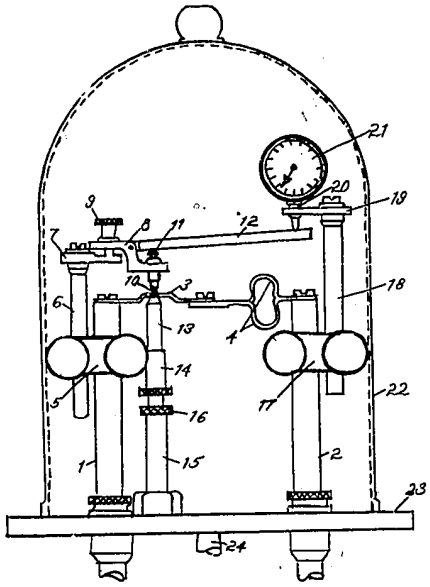

Figure 5. Device for Hardness Testing of the Specimens on Heating in Vacuo

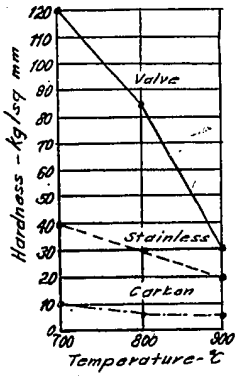

Figure 6. Changes in Hardness of Valve Austenitic, Stainless Chrome-Nickel, and Carbon Steels on Heating to 900° in Vacuo

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